

Exhibit 1

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Chemical Engineers' Handbook

FIFTH EDITION

Prepared by a staff of specialists
under the editorial direction of

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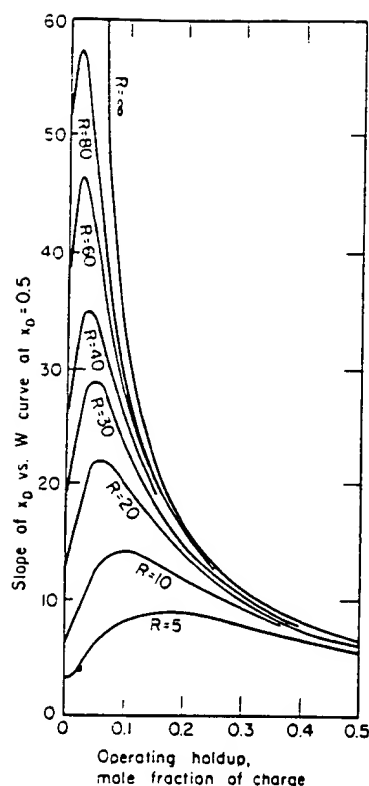


Fig. 13-61. Sharpness of batch separation as function of operating hold-up and reflux ratio (L/D) for cyclohexane- n -heptane equimolar mixture in 25 theoretical-plate columns. [Houtman and Husain, *Chem. Eng. Sci.*, 5, 180 (1956).]

2. As in the McCabe-Thiele graphical method, each plate has a horizontal and a vertical segment. The horizontal segment starts with a known y composition. The x composition for the plate is found by interpolation of the equilibrium data between points which bracket the starting y value. For the vertical segment, the intersection is found for the x value and the operating line. The equation of the intersection is $y = (y_D + Rx)/(R + 1)$. The horizontal-vertical process is continued for the number of plates assumed in the column.

3. For the constant-reflux case, an arbitrarily high trial initial-distillate composition is chosen, and the pot composition is found through a plate-to-plate determination. A comparison is made with the composition of the charge. If it is higher, the initial-distillate composition is reduced incrementally. Once the calculated pot composition drops to the charged value, distillate and pot compositions are accumulated for successively lower distillate values. They

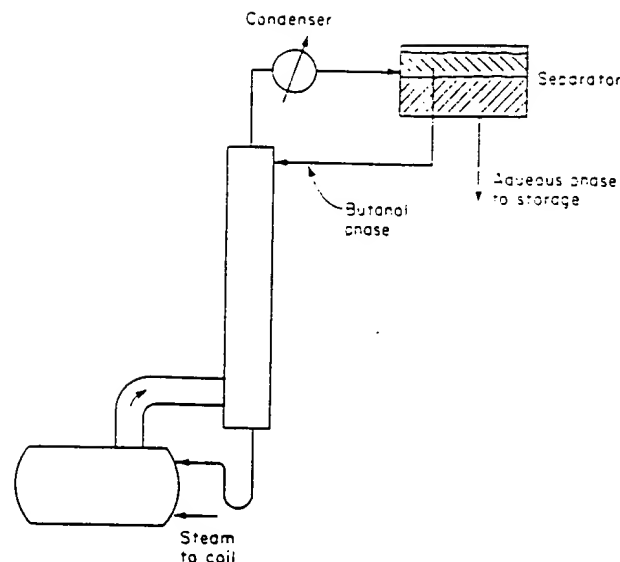


Fig. 13-62. Azeotropic distillation of water from n -butanol in a batch column.

may be integrated as the distillation proceeds, or they may be stored and the integration performed when the process is concluded. The end of calculations may be signaled by an arbitrarily low pot composition or by the specification of an average distillate composition.

4. In the case of constant composition the procedure is similar. In this case the distillate composition stays the same and the slope of the operating line changes. Since the distillate is not changing, a convenient end point is some arbitrarily high reflux value.

For further reference, an analog approach to computer solution was taken by Bowman and Clark [*Chem. Eng. Progr.*, 59, 54 (1963)], and Robinson and Goldman [*Brit. Chem. Eng.*, 14, 509 (1969)] utilized a digital simulation technique.

Azeotropic Batch Distillation. Batch columns are used extensively for azeotropic separations. Instead of using multiple columns, cuts are segregated in storage for later reuse or distillation. In the separation of n -butanol-water, for example, the process starts by charging the original mixture into the still pot (Fig. 13-62). In the first distillation the water phase is accumulated in a storage tank for later processing. The second cut is an intermediate mixture rich in butanol with the remaining traces of water. It is also stored to be added to the next fresh batch of feed. The remaining liquors are essentially pure n -butanol, but they may also be distilled to remove impurities. If so, the final heel of material is added to successive batches until sufficient impurity builds up to warrant purging. The accumulated water phase from several batches is distilled. The butanol-containing distillate is added to incoming material, and the remaining water in the pot is purged.

MOLECULAR DISTILLATION

Definitions. Three broad classes of high-vacuum distillation can be discerned (see Table 13-14): (1) *conventional* apparatus, generally consisting of a boiler, fractionating column, and condenser, operated under high vacuum; (2) *unobstructed-path* distillation operated under high vacuum; and (3) *molecular* distillation, where the vapor path is unobstructed and the condenser is separated from the evaporator by a distance less than the mean free path of the evapo-

Table 13-14. High-vacuum Manometric Range

Class of still	Laboratory size, mm.	Industrial size, mm. Hg
1. Conventional stills	0.01-0.1	0.1 -5.0
2. Unobstructed-path stills	0.01-0.1	0.001 -0.01
3. Molecular stills	0.001	0.0001-0.001

rating molecules. This section deals with categories 2 and 3; "high" vacuum is defined as a pressure of residual gas so low that further reduction does not change the performance of the apparatus.

Molecular and short-path distillations are generally conducted at pressures of 1 to 7μ , rather than 1μ being readily available in

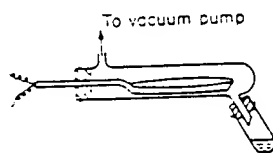


Fig. 13-63. Simple molecular still.

the laboratory, whereas about 3 to 30μ have proved economical in industry (μ = micron = 0.001 mm. Hg). The differences between unobstructed-path and molecular distillation lie in dimensions and operating conditions; thus one piece of apparatus can be either a short-path or a molecular still, according to the rate of distillation and the pressure of residual gas.

Simple Molecular Still. A simple and practical molecular still with an evaporative efficiency of about 0.5 is formed by an electrically heated tray suspended in an evacuated test tube (Fig. 13-63). An evaporative efficiency of 0.5 means that 50 per cent of all the molecules evaporating are able to leave the containing cup at first try and reach the condenser to be completely condensed without re-evaporation.

An all-glass molecular pot still is shown in heavy outline superimposed on a conventional still and condenser in Fig. 13-64. Heat is applied beneath the still, and the ceiling is cooled by ice or a blast of air. Liquid condenses on the ceiling and drops into the receiver. Considering any typical element of liquid surface S , a molecule can evaporate in any direction embraced by a solid angle of 180 deg. Those molecules which proceed within the wide cone m, s, m' will condense on the ceiling and be collected; the rest will condense on the walls and return to the still. Contrast this with the conventional still where only those molecules within the narrow cone d, s, d' will pass up the neck of the flask, and only an exceedingly small fraction s, r, r' will wander into the receiver.

Ordinary laboratory "vacuum" distillations are done at 1 to 10 mm. Hg in order to transfer the vapor rapidly enough to complete the distillation in reasonable time. The efficiency of transfer of the molecular pot still of Fig. 13-63 is 0.4 to 0.6; that of the conventional still is 0.001 to 0.0001. Stated another way, an evaporating molecule is likely to escape permanently at the first or second try in a molecular still, whereas it may return to the distilland a thousand times before finally reaching the exit of an ordinary vac-

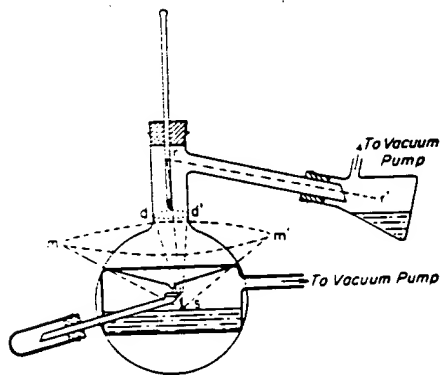


Fig. 13-64. Molecular pot still, superimposed on conventional still and condenser.

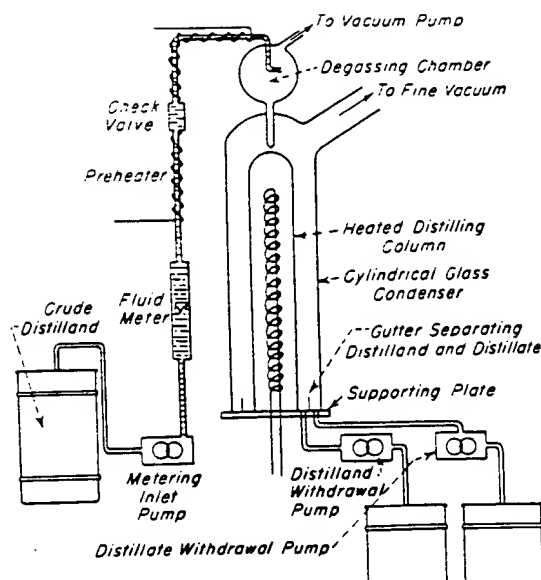


Fig. 13-65. Falling-film molecular still.

uum still. Temperature of distillation and hazard of decomposition are reduced in proportion to the lowered pressure of the molecular still.

Mean-free-path Considerations. The definition of molecular distillation as "within the mean free path of the emergent molecules" implies that, if the molecules suffer collision of any sort, particularly with molecules of residual gas, they will be delayed in reaching the condenser or may return to the distilland; distillation will be slowed and will become progressively more equilibrant in kind. Burch showed how the chance that a molecule will reach the evaporator at one strike should vary with the distances, in multiples of the free path. Cox and Hickman measured the change in rate of distillation with change in pressure of residual gas and found that collisions mattered less than formerly supposed. The comparison of pressure, free path, and rate of distillation is given in Table 13-15, which shows that the rate is diminished by less than half even though not more than one molecule in 20,000 reaches the condenser without collision.

There are three important calculations of the free path L :

$$\text{Maxwell's: } L = \frac{1}{\frac{4}{3}\pi\sigma^2 N} \quad (13-157)$$

$$\text{Clausius': } L = \frac{1}{\sqrt{2}\pi\sigma^2 N} \quad (13-158)$$

$$\text{Tait's: } L_T = \frac{0.677}{\pi N \sigma^2} \quad (13-159)$$

Table 13-15. Comparison of Pressure, Free Path, and Rate of Distillation

Pressure of residual air in 2-cm. gap, μ	2-cm. gap is approximate multiple m of free path	Comparative rate of distillation, %, with saturation pressures of		Number of molecules, Φ , reaching condenser without collision (Burch)
		1μ	10μ	
0.3	$m = 0.2$	100	100	80
4.0	$= 3$	77	89	
7	$= 5$	63	81	0.005
10	$= 7$	53	72	
25	$= 17$	35	42	
50	$= 33$	20	27	

where N is diameter of molecule σ as

in appropriate gas molecules used paths and a distill. and 2 cm. f

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where N is the number of molecules in unit volume and σ the diameter of the molecules. The mean free path L_{12} of a large molecule σ_2 , distilling into a permanent gas σ_1 , is given by Loeb as

$$L_{12} = \frac{1}{\sqrt{2} \pi N_2 \sigma_2^2 + \pi N_1 \sigma_1^2 \sqrt{\frac{C_1^2 + C_2^2}{C_2}}} \quad (13-160)$$

in appropriate units, where C_1 and C_2 are mean speeds of residual gas molecules and evaporating molecules, respectively. The measured paths of heavy molecules in a residual air vacuum of 1μ and a distillation pressure of 1μ are about 3 cm. for butyl phthalate and 2 cm. for olive oil.

The Distilland Layer. It is assumed in ordinary distillation that the surface of the distilland is at any moment a true sample of the liquid, that diffusion of volatile molecules to the surface of a liquid mixture is rapid compared with their rate of evaporation. This condition is demonstrably unfulfilled in the molecular still, where convection due to ebullition is absent and high viscosities and high molecular weights impede diffusion. Efficient molecular distillation requires mechanical renewal of the surface film. This can be done in three ways: (1) by vigorous agitation of the liquid in bulk, i.e., the stirred pot still; (2) by gravitational flow in cascade or vertical falling film; or (3) by mechanical spreading in thin films. The last is accomplished by transferring the distilland to a heated roller or traveling disk, cone, or cylinder. The heated rotating cone and the wiped cylinder are currently the preferred forms of evaporator.

Commercial Apparatus. Falling-film stills have been made in single and multiple units, in sizes ranging from a few centimeters to 50 cm. diameter, 2 to 10 meters high, with through-puts ranging from 1 to 60 liters/hr. The elements of construction are shown in Fig. 13-65. The distilland is admitted through a metering device into the vacuum where it is degassed in one or more preliminary vessels and then allowed to pass on to the walls of a heated polished metal cylinder stationed within a concentric cooled condensing cylinder. The space between the two is maintained under high vacuum (1 to 5μ) by an oil-filled diffusion pump backed by a mechanical vacuum pump or a multistage steam ejector.

Centrifugal stills, shown diagrammatically in Fig. 13-66, consist of a housing or base plate covered by a lid or dome which serves as the air-cooled condenser. A flat or conical plate on which the distilland will be spread by centrifugal force is supported on a shaft that passes through a bearing and stuffing box attached in the base plate. A radiant electrical heater warms the plate, and radiation is conserved by baffles placed at the back and sides of the heater. Distilland is admitted from a preliminary degasser through a feed pipe to a depression at the center of the rotor, whence it is spun rapidly outward in an exceedingly thin, uniform layer. At the edge, the distilland, spent after evaporation of the volatile constituents,

is picked up by a scoop (Fig. 13-67) or collected by a concentric gutter.

Since the distilland is held on the evaporator by centrifugal force, the operation is independent of gravity, and the rotor may be horizontal or vertical or even upside down. A convenient angle

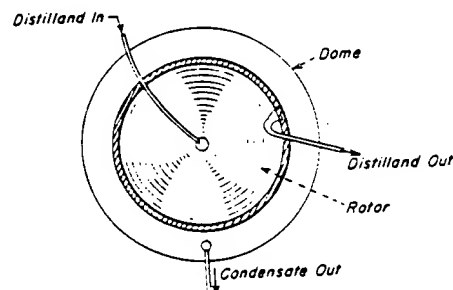


Fig. 13-67. Distilland flow in centrifugal still.

for small stills (12 to 36 in. diameter) is with the evaporator facing upward at about 45° . The rotors of larger stills are generally cones instead of plates, and they spin on vertical shafts. The condenser hangs within the cone, and means are provided for pumping the condensates, collected separately from three concentric zones, over the rim. Stills of this type have evaporating areas of about 4.5 sq. meters and handle 200 to 700 liters of distilland per hour, collecting 2 to 400 liters of distillate per hour, according to the object of treatment. The consumption of power is about 100 kw., 60 per cent for heating the still, the remainder for pumps and subsidiary equipment.

In the last few years the falling-film still has been improved greatly by the addition of wipers. The inside of the vertical tubular container is made the evaporator and the inner concentric tube the condenser. Carbon or Teflon wipers, held in a cylindrical cage, are rotated between evaporator and condenser and agitate the distilling surface many times each minute. Because of a very slight splashing, it is sometimes useful to place a porous baffle between the two surfaces and rotate it with the scrapers. The general design is in increasing use for petrochemicals and in heavy industry. An example of a wiped still is shown in Fig. 13-68.

Distillation Efficiency and Rate. It is a safe generalization that all types of unobstructed-path stills provide non-equilibrant distillation. In ordinary distillation of a mixture, the quantities of constituents distilling are proportional to their partial pressures P_1, P_2, \dots, P_n , but, under molecular conditions, the quantities are

$$\frac{P_1}{\sqrt{M_1}}, \frac{P_2}{\sqrt{M_2}}, \dots, \frac{P_n}{\sqrt{M_n}}. \text{ Since substances of like molecular}$$

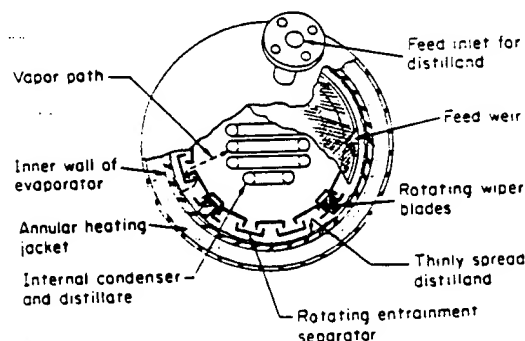


Fig. 13-68. Plane horizontal cross section of a commercial wiped-film molecular still. (Pfaunder-Permutit section, Sybron Corporation, Rochester, N.Y.)

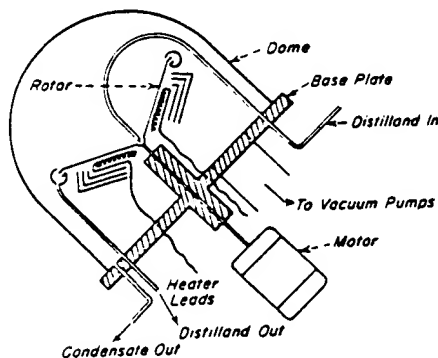


Fig. 13-66. Centrifugal molecular still.

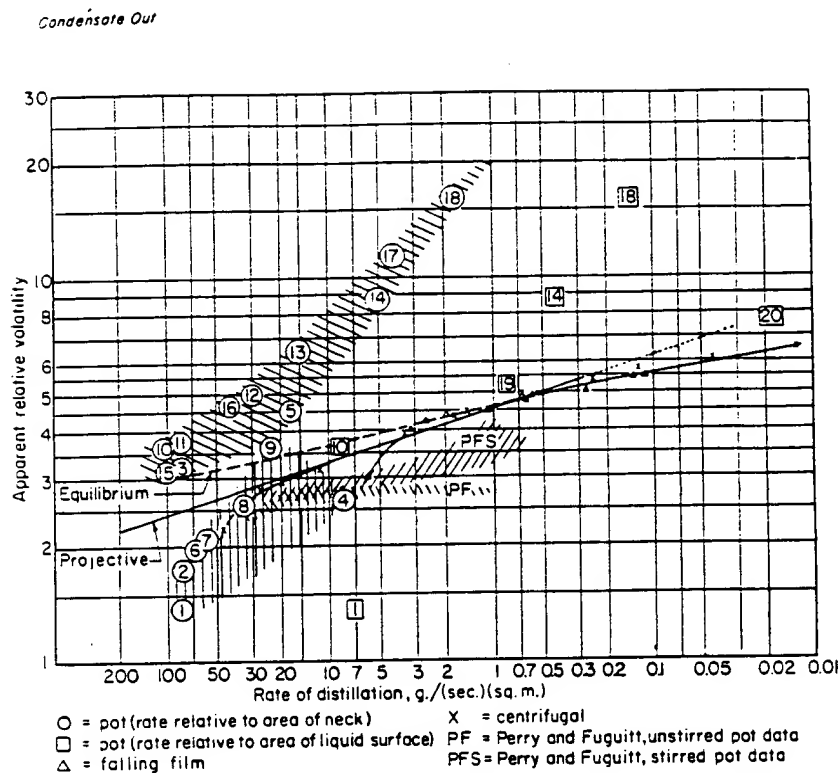


Fig. 13-69. Separatory power of various stills as a function of rate. [Hickman and Trevo, *Ind. Eng. Chem.*, 44, 1908 (1952).]

weight distill at similar temperatures in natural mixtures of organic substances (e.g., plant or animal fats, sterols, or hormones) the kind of separation available from molecular distillation scarcely differs from equilibrant distillation. It is the degree of thermal exposure that is so markedly less in the molecular still.

The maximum degree of separation available in a single pass through a short-path still is "one theoretical molecular plate." Much argument and research have been devoted to defining this unit. It has become obvious that the unit is variable, dependent on conditions, being larger at slow rates of evaporation and most nearly approached when there is great surface agitation. Hickman and Trevo [*Ind. Eng. Chem.*, 44, 1908 (1952)] compiled a map (Fig. 13-69) of the behavior of two test fluids, 2-ethyl hexyl sebacate and 2-ethyl hexyl phthalate, in "ideal" high-vacuum stills and the practical stills of the laboratory. Centrifugal, falling-film, and stirred-pot stills all gave optimum separations, defined as one theoretical plate, at absolute rates of evaporation corresponding to vapor pressures of 0.005 mm. Hg or less, but only the centrifugal still yielded near optimum separation at the more useful rates of 0.01 to 0.1 mm. Hg, e.g., in the yield range of 5 to 50 kg./hr.(sq. meter). The other types gave useful but lesser separations. The circled numbers of Fig. 13-69 refer to still constructions in the original paper.

The poor separations resulting from a simple act of molecular distillation mean that, in a series of distillates removed from a complex mixture, any given constituent will appear in *some concentration* in every fraction. If the fractions are withdrawn at equal time intervals and uniform increments of temperature, the variation of yield of a component A in the distillates follows a smooth probability curve, and this applies also to components B, C, . . . , N, so that the concentrations of each overlap according to the scheme shown in Fig. 13-72. The position of the maxima of these curves on the temperature axis, obtained under rigidly standardized condi-

tions, provides the molecular substitute of a boiling point and one that can be made accurate, or at least reproducible, within $\pm 1^\circ\text{C}$.

The quantitative rate of distillation, under free-path conditions of evaporation and complete non-return condensation, is equal to the quantity of material passing across an imaginary area in a saturated vapor of the distilland equal to the area of the evaporating surface and is given numerically by the Hertz-Knudsen equation

$$w = 0.0583p \sqrt{\frac{M}{T}} \quad (13-161)$$

where w = distillation rate, grams/(sec.) (sq. meter of evaporating surface); p = saturation pressure, mm. Hg, at the solution temperature T °K.; M = molecular weight. For the kind of substances handled in molecular stills, $w = 0.5$ gram/(sec.) (sq. meter). Small though this figure may appear, it means that two stills 5 ft. in diameter can process a tank car of oil in 24 hr. A laboratory falling-film still can subject a sample of 200 cc. to 15 to 20 passes in half a working day.

Coefficient of Evaporation. Although the highest possible rate of evaporation for a specified substance, temperature, and vapor pressure is given by the Hertz-Knudsen equation, it is common experience that such rates are seldom achieved. So universal is this default at higher than "molecular" pressures, e.g., > 10 mm. Hg, that the concept of a coefficient of evaporation E , far less than unity, became prevalent in the period 1925-1930; a liquid was credited with reflecting molecules of its own vapor instead of condensing them at first impact. The property was supposed to reside in polar molecules, hydroxylated compounds, and especially glycerin and water, the latter being assigned $E = 0.03$ to 0.05.

Measurements made by chemical engineers (Bromley, Nabavian, Mills, Jamieson, *et al.*) and by experimental chemists (Hickman and many others) point strongly to a coefficient of unity $E \sim 1$ for all clean liquid surfaces, a conclusion supported by Trevo's (1954)

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distillation of glycerin with $E = 0.9$ to 1.0 over a wide range of vapor pressures; by Hickman's evaporation of cold water at $E < 0.5$ (1965), and Maa's many calculations of surface temperatures (1967) showing that $E = 1.0$ for the clean water-vapor interface. Two barriers lower the practical coefficients \bar{E} of liquids—repressive monolayers of impurity molecules and much micro- and macroscopic debris; and the outermost laminae of the liquid boundary layer (the donor-conductive layer, Hickman, 1965) which limits the flow of heat for evaporation. Both require continuous and rapid displacement by natural means (e.g., Langmuir "twitching," 1927) or mechanical devices (stirring, centrifugally driven or wiped films, 1930-1960). Raising the apparent evaporation coefficient \bar{E} is thus an ever-present concern of the chemical engineer.

Subsidiary Apparatus. Molecular stills require high-vacuum pumps in order to operate. Since the pressure in the still should be $< 10^{-5}$ of an atmosphere, and since no practical pump can achieve this reduction of pressure in one stage, it is usual to employ two or more pumps in series, each unit being designed especially for its place in the series. A solitary exception is the laboratory micro-pot still, which can be evacuated sufficiently well by a rotary oil pump (8 to 12 μ in the still).

The pump that is used for the ultimate vacuum is by general choice the Langmuir condensation pump. The laboratory falling-film still, handling one liter/hr. of distilland, requires a condensation pump with a capacity of 100 liters/sec. at 1 to 2 μ , which gas it will pass on to a mechanical forepump at a pressure of 0.1 to 0.2 mm. The forepump must have an effective volumetric capacity of at least 1 liter/sec. If it is not desired to employ such a large mechanical pump, a vapor booster pump may be inserted between the condensation pump and the mechanical pump. The booster will take in gases at 0.2 mm. and 1 liter volume, compressing them to 0.5 mm. and 0.4 liter volume, which can be handled by the smallest commercial pump.

Industrial centrifugal stills can be operated by large condensation pumps in series with oil-sealed rotary mechanical pumps, capacity 100 to 200 cu. ft./min. The materials being distilled are almost always contaminated with impurities that are volatile at the temperatures and pressures prevailing in the still but condense to liquids under the conditions in the mechanical pump. The sealing fluid soon becomes fouled, the impurities re-evaporating to fill the suction space at each intake stroke of the pump and condensing again on each compression stroke. The volumetric capacity of the pump decreases to a small fraction of optimum. Furthermore, the contaminants, especially fatty acids, are likely to be corrosive, causing the rotor to jam after a few hours' use. Thus industrial stills employ steam ejectors as prime movers, which often have the advantage of flushing the odors away into the chimney or drain. A typical pumping train for a large still will consist of three stages of steam, two oil boosters, and a condensation pump of the self-fractionating or purifying variety, capacity 1000 to 5000 liters/sec., next to the still.

The molecular still also requires inlet pumps and meters for the distilland and withdrawal pumps for the distillate. All must be of a kind that will not "vapor-lock" or "overrun" in passing from atmospheric pressure to vacuum.

The final and major requirement is heat—now by common usage electrical heat. Because distillation is done at high temperature

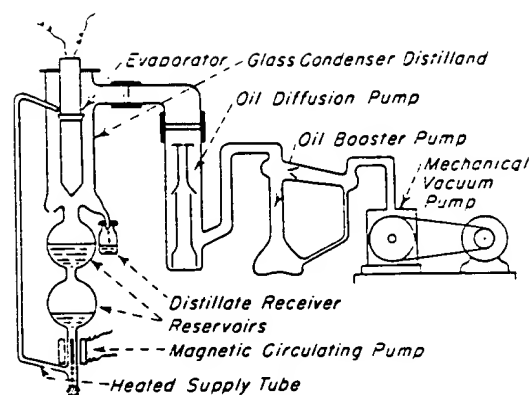


Fig. 13-71. Cyclic batch falling-film still.

(200° to 300°C.) and must often be repeated to secure proper separations, and because the hot evaporator loses heat by radiation to the cold condenser, less than 5 per cent of the input heat is actually used by distillation. When the heat required for the oil-vapor vacuum pump is also debited against the electrical consumption, the thermal efficiency may fall to 2 to 3 per cent. However, the through-put of the modern molecular still is sufficiently high for the total operating cost to compare favorably with conventional distillation.

Uses of the Molecular Still. Laboratory. The small demountable pot still (Fig. 13-70) is an intermediary step in the estimation of vitamin E in foods and oils. The same apparatus is useful for purifying small samples of drugs, dyes, sterols, and hormones. For the investigation of natural oils and waxes, the cyclic batch falling-film still is generally employed (Fig. 13-71). This instrument is designed for multiple repasses of the distilland under standard conditions of increasing temperature. The investigator can construct an *elimination curve* of materials under investigation which affords a precision method of analysis (Fig. 13-72). The cyclic batch centrifugal still has been developed in small size, convenient for bench-top use (Fig. 13-73).

Industrial. The falling-film still has largely been replaced by centrifugal units. These have evaporators approximately 1, 3, or 5 ft. in diameter and are generally grouped in blocks of three to seven to allow fractionation by multiple redistillation. Their chief uses are distillation of vitamin A esters and intermediates, stripping of vitamins E (α -, β -, γ -, and δ -tocopherols) and sitosterols from vegetable oils, and the complete distillation of industrial high-boiling synthetics, plasticizers, fatty acid dimers, and the like.

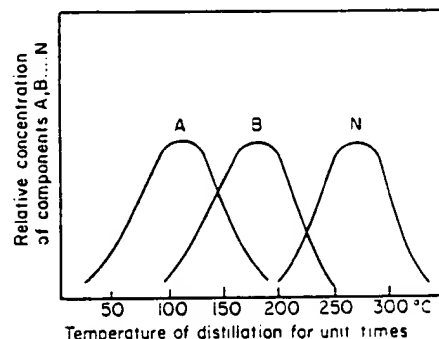


Fig. 13-72. Molecular distillation of components A, B, ... N in a mixture.

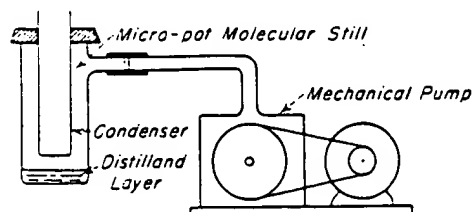


Fig. 13-70. Demountable pot still.

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(13-161)

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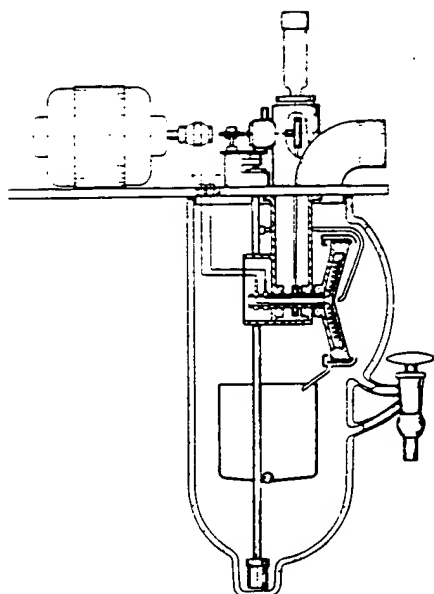


Fig. 13-73. Cyclic batch centrifugal still.

Recent uses are for separating mono- and di-glycerides for bread and paraffin wax for milk cartons.

Comparative Properties of Molecular Stills. Modern high-vacuum stills perform distillations at what are believed to be the lowest theoretically possible temperatures, and they accomplish this in the shortest times at present attainable in distillation equipment. For example, even an industrial flash distillation done at 10 mm. Hg involves a thermal exposure about 300,000 times greater than that caused by a high-speed molecular rotor. Against these advan-

tages of the molecular still must be placed the poor separatory power of the unit act of distillation.

Fractionation. Separations better than unity—one molecular plate—can be secured by a series of redistillations done in a cascade of separate molecular stills. If the feed of a binary mixture is

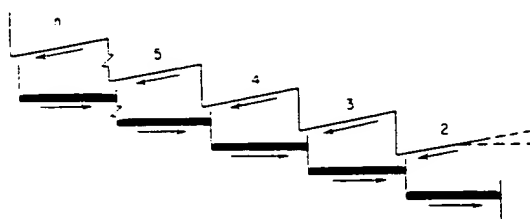


Fig. 13-74. Multicompartment molecular still.

admitted at approximately the center of the cascade, and if distillates are blended with the residues one still backward and the residues with the distillates one still forward, purified components emerge at either end of the cascade. Two self-contained units have been described for doing this, the laboratory glass still of Wollner and associates and the steel 10-compartment still of Brewer and Maddorski (Fig. 13-74), which was devised for separating the isotopes of mercury.

The multiple fractionator of Fig. 13-74 is redundant in that the surface of distilland in each compartment remains unstirred so that the separation coefficient α , for the components of a mixture, analogous to E , the coefficient of rate for a single component, may remain far below unity—what could be gained by repetition is partly lost by inefficiency, while the *thermal hazard* (Embree, Hickman, 1948) is multiplied by the number of steps. The situation has been remedied in various ways, e.g., Perry (Brush still, 1946) and Ridgeway Watt (1965), who introduced a rotary modification of Brewer and Maddorski's stationary cascade (Fig. 13-74). A catalog picture of Watt's device (Bendix Vacuum, Rochester, N.Y.) is included as Fig. 13-75. The efficiency of each stage, tested by separation of EHP from EHS, is said to exceed $\alpha = 0.9$.

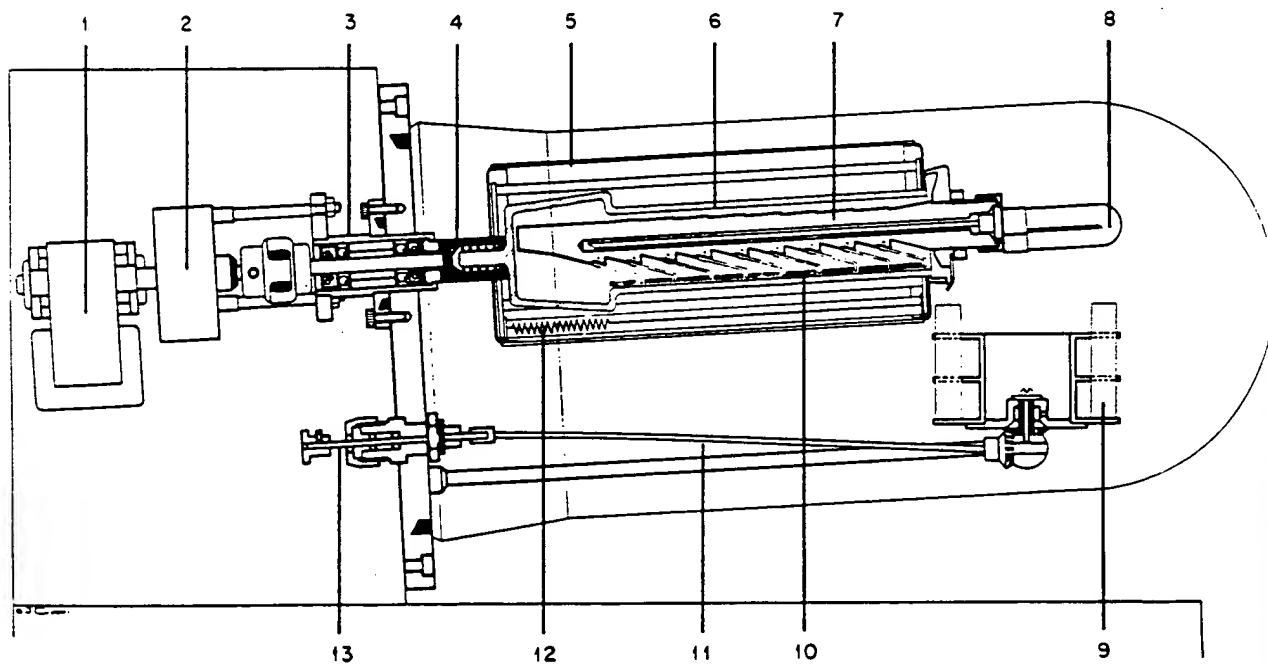


Fig. 13-75. A rotary multistage molecular still.

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